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Change in particle size distribution of Torrefied biomass during cold fluidization

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ABSTRACT

Torrefaction is a thermo-chemical process for upgrading biomass that is usually run at temperatures ranging from 200 to more than 300 °C in an oxygen-free atmosphere and at ambient pressure. It is a useful pre-treatment technology for biomass that significantly alters its physical and chemical composition, making it more efficient in pulverized systems. This paper investigates the grindability of eucalyptus chips torrefied at temperatures ranging from 210 °C to 270 °C for a 15 min residence time, compared to raw material before and after fluidization. The tests were carried out in a cold flow fluidization bed chamber with an internal diameter of 150 mm, with air as the fluidizing medium and a chamber height of up to 750 mm. Biomass grindability was assessed by evaluating the change in particle size distribution. The results showed that the degree of thermal degradation depended on the temperature and was also influenced by particle size. The mean particle size of ground torrefied biomass decreased with an increase in torrefaction temperature. Lastly, for larger particles, it was possible to correlate the size distribution with the severity of the heat treatment by a simple linear equation in our experimental conditions.

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1. Introduction

Biomass can be used to produce energy by different technologies: thermochemical (combustion, gasification), biological (anaerobic digestion, fermentation) or chemical processes (esterification). Due to its low energy density compared to fossil fuels, very high volumes of biomass are needed, which compounds problems associated with storage, transportation, and feed handling at cogeneration, thermochemical, and biochemical conversion plants. Torrefaction, which is a thermal pretreatment process [1] is an economically viable technology that significantly alters the physical propertiesand chemical composition of biomass making it more efficient in pulverized systems, such as for co-firing and gasification purposes [2]. Most of the technology has been developed and applied industrially for wood preservation in recent decades [3]. Properties such as a high energy density and high mechanical fragility are now expected. The reader will find a detailed literature review in Ref. [4].

Pilot Plant testing with different biomass types has already shown that the feedstock flexibility of the current generation of torrefaction technology is limited. Further R&D efforts are needed to torrefy various type of biomass (wood and agricultural waste) in a technically and economically viable way. There is a need to optimize the balance between particle size, temperature and retention time, which vary depending on the type of biomass [5].

The chief advantage of fluidization is that the solid is vigorously stirred by the fluid passing through the bed; however, biomass particles have unusual properties which make them difficult to fluidize and handle because of high cohesion forces between the particles and channelling [6]. Previous studies on biomass fluidization have focused on obtaining hydrodynamic properties such as particle size, diameter, bulk density, fluidizing velocity, etc. [7]. According to Gildart's distribution diagram classifying biomass residue fuel, bulk density and voidage are found to be the main factors contributing to the fluidizing quality of the bed [8]. Other authors have studied changes in the cold fluidization behaviour of torrefied biomass. Rousset [9] determined minimum fluidization velocity and compared it with theoretical values. Depending on the processing conditions and particle size of raw biomass, the tendency showed a greater pressure drop for untreated biomass than for treated biomass at the same velocity. Raw biomass has poor grindability. After torrefaction, grindability characteristics are improved when compared to raw biomass; the biomass is weakened, which results in a reduction in pressure drop.

The level of biomass destruction can be determined by sieving and analysing the size distribution of the fragments. The originality of this work consists in studying changes in the friability and

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Table 1
Characterization of *E. Grandis* chips (group M). GCV = Gross Calorific value.

Bulk density (kg m^{-3})	Volatile matter (%)	Fixed carbon (%)	Ash (%)	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Oxygen (%)	Lignin (%)	CGV (j.g^{-1})
490	80.15	19.56	0.29	51.2	5.8	0.05	42.95	35.6	18030

particle size distribution of torrefied *Eucalyptus* sp. at 210, 240 and 270 °C for 15 min and further cold fluidization. An analysis of variance was used to test for particle size and treatment group differences.

The findings in this study could be used to define appropriate torrefaction parameters for optimized biomass particle size prior to use in a pulverized system.

2. Material and method

Eucalyptus sp. chips from a sawmill were used as the raw material. Table 1 gives the main physical, chemical and energy properties of the biomass. The samples used to study fluidization behaviour were dried beforehand at 103 °C for 48 h and heated to selected temperatures (210, 240 and 270 °C) for 15 min in an inert atmosphere using a lab-scale torrefaction reactor as described in Ref. [10]. The system provided continuous acquisition of the temperature, oxygen level and sample mass. The data were recorded every minute during thermal decomposition.

Lastly, the aim was for the temperature history of the wood samples to be very close to that of the set temperature acting on the gas flow. This was ensured by a high heat transfer coefficient at the interface and by choosing a small sample thickness to reduce the time constant related to thermal diffusion in wood. This time constant τ read as: $\tau = (t^2 \rho C_p)/\lambda$ where t is the half-thickness of the sample (m), ρ the wood density (kg m^{-3}), Cp the specific heat capacity of wood (J kg^{-1} K^{-1}) and λ the thermal conductivity of wood (J s^{-1} m^{-1} K^{-1}). Using usual values for wood (ρ = 800 kg m^{-3}, C_p = 1250 J kg^{-1} K^{-1} and λ = 0.15 J s^{-1} m^{-1} K^{-1}) and with a total thickness of 1.10^{-6} m, the time constant τ was less than 16 s. This value was very small relative to the treatment duration, thereby ensuring uniform treatment throughout the section.

The fluidization tests were carried out using a lab-scale cold fluidized reactor as described in Refs. [9], consisting of a batch type plexiglass fluidizing column of approximately 750 mm in length with an internal diameter of 150 mm, in which the biomass was placed for fluidization and observation. Air was provided by a blower (a 0–60 Hz synchronous electric motor) coupled to the fluidizing column by flexible ducts, from which the outflow was controlled by a frequency inverter with a programmable output frequency. Biomasses were fluidized for 20 min (1 min for each frequency selected) varying the air velocity from 0 to 3 m s^{-1} as a function of frequencies in the column. The bed height for each test was 10 cm, corresponding to an average mass of 450 g of biomass (Fig. 1). Lastly, wood chips were passed through different sieves to analyse the impact of torrefaction and fluidization on particle size distribution. Fig. 2 sums up the experimental protocol used in this study.

Wood chips from the raw material were passed through different sieves to obtain the appropriate particle size before the experiments. Four particle sizes in mass proportion were obtained from the mixture denoted M (Fig. 3). It has been shown that torrefaction decreases both grinding energy and particle size distribution [11]. The energy required to obtain a fine powder is proportional to the particle size distribution [12]. For the rest of this study, two groups of particles were ruled out: those that were not representative (X_3 under 3%) and those not requiring grinding ($X_0 < 0.28$ cm) as they had acceptable flowability through the processing and transportation systems [13]. The fluidization study was thus carried out on the following three size groups called M, A and B:

M comprising X_0(34.8%), X_1(18.6%), X_2(44.2%) and X_3(2.4%)
A comprising X_1(100%)
B comprising X_2(100%)

3. Results and discussion

3.1. Mass loss

Table 2 gives the gravimetric yields γ versus temperature for each particle size group (M, A and B). Gravimetric yield (γ) is the ratio of torrefied wood mass to initial feedstock mass, dry at 0%. The tendency revealed mass loss increasing with temperature. Exposure of biomass to high temperatures results in thermal degradation of its structure, which is often accompanied by loss of mass. The degree of thermal degradation depends on the heating time and temperature, and is also influenced by particle size [14]. At 210 °C, this temperature zone, also called the reactive drying range, initiates the breakage of hydrogen and carbon bonds. Mass loss predominantly results from the loss of moisture, and the depolymerization of hemicelluloses results in shortened, condensed polymers with solid structures [15]. Up to 240 °C, hemicelluloses decompose extensively into volatiles and a char-like solid product. Lignin and cellulose show limited devolatilization and carbonization. Gravimetric yield was found to be lower for smaller particle sizes: 90 and 84% for A at 240 and 270 °C respectively, and 94 and 88% for B. These observations could be explained by the fact that heat and mass transfer was facilitated for small particles, as

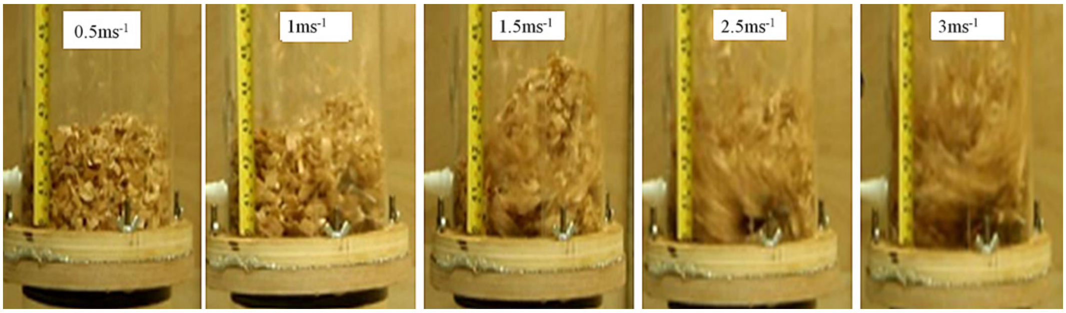

Fig. 1. Visual observations of raw biomass fluidization for a 10 cm bed height. Full fluidization starts at around $V = 2.5$ m.s^{-1} ($F = 40$ Hz) upwards.

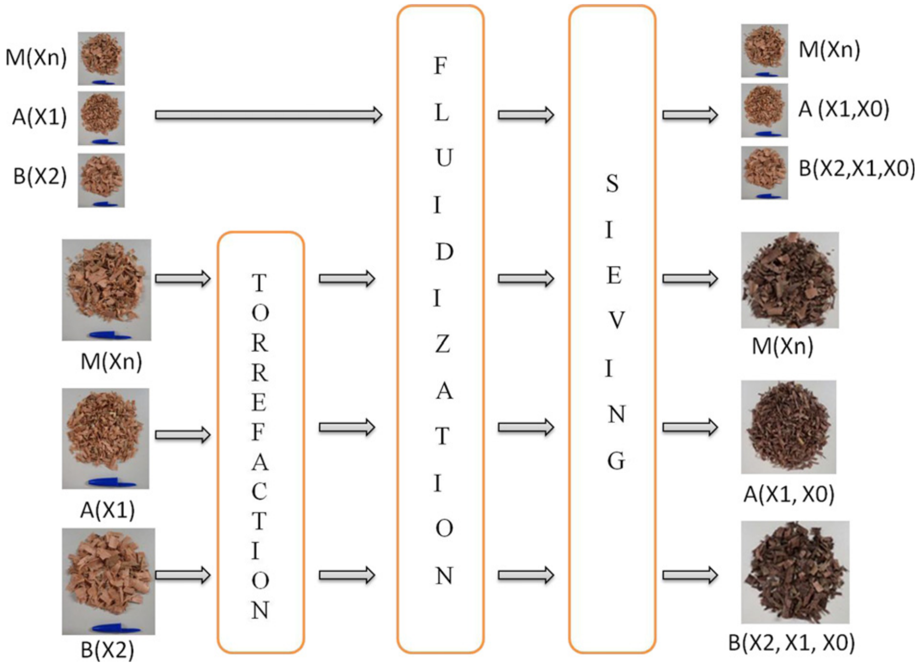

Fig. 2. Typical particle size distribution of Eucalyptus chips at various screen sizes.

demonstrated in Ref. [16]. The biomass mixture group denoted (M) had a behaviour somewhat similar to group B, whereas A was more reactive, as displayed in Fig. 4. Almeida and al. [17] proposed overall mass loss as a relevant parameter for synthesizing the effect of torrefaction conditions (temperature and duration).

Fig. 4 shows the mass loss trend as a function of time for the treatment at 270 °C, along with the differential thermal gravimetry expressed in g min^{-1}, calculated from the derivative of the mass loss curves over time. For easier reading, the curves are not given for 210 and 240 °C respectively, but the shapes were similar. In all cases, the tendency revealed an increase in maximum mass loss rate as the temperature increased in the reactor. Maximum reactivity for the three groups occurred at 150 min when the temperature in the reactor reached its maximum, i.e. 270 °C. It was found that the maximum mass loss rate was 35% higher for A compared to B and M. Group A was composed of the smallest particles compared to B and M. At equivalent temperature, heat transfers are facilitated when particles are smaller, leading to a higher mass loss rate, as can be seen in this figure. These results corroborate previous work [18]. Some previous work assessed the improvement of pulverized torrefied biomass. Chen [19] indicated that when the torrefaction temperature is relatively low, such as 230 and 260 °C, the weight loss of biomass significantly depends on the temperature, as a result of hemicellulose and cellulose thermal degradation. However, once the torrefaction temperature is as high as 290 °C, the weight losses of various biomass materials tend to become uniform.

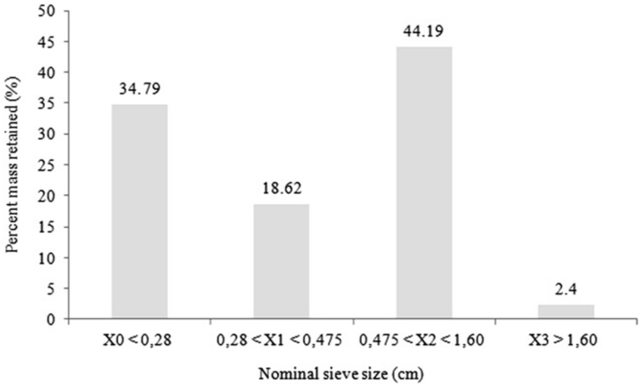

Fig. 3. Experimental protocol; X_n where $n = 1$ to 4.

3.2. Particle size distribution

Particle size distribution curves are an important parameter for understanding flowability and combustion behaviour during co-firing. Biomass is highly fibrous and tenacious in nature. During torrefaction, biomass loses its tenacious nature resulting in decreased fibre and particle length, making it easier to grind and pulverize [20]. In order to assess the impact of fluidization on particle size distribution prior to torrefaction, several fluidization tests were carried out with the initial non-heat treated raw material (group M) representing the whole set of particles X_0, X_1, X_2 and X_3. Particle size distribution measurements were taken before and after fluidization on the reference sample in Fig. 2. Each run was repeated three times. The results were statistically processed by an analysis of variance using XLSTAT statistics and data analysis software [21]. Table 3 gives the coefficient of determination R^2 and the P-value for each group of particles. It can be seen that only particles over 1.6 cm (X_3) were affected by fluidization with a coefficient of $R2 > 0.99$ and a P-value < 0.05. The smallest particles did not undergo any statistically significant modifications before and after fluidization. The ability of biomass to resist mechanical efforts depends on particle size, among other things. A large particle will be

Table 2
Gravimetric yield γ (%) for each particle size group as a function of temperature.

Temperature	γA (%)	γB (%)	γM (%)
210 °C	98.47	97.86	97.13
240 °C	90.17	94.63	95.02
270 °C	84.62	88.00	87.14

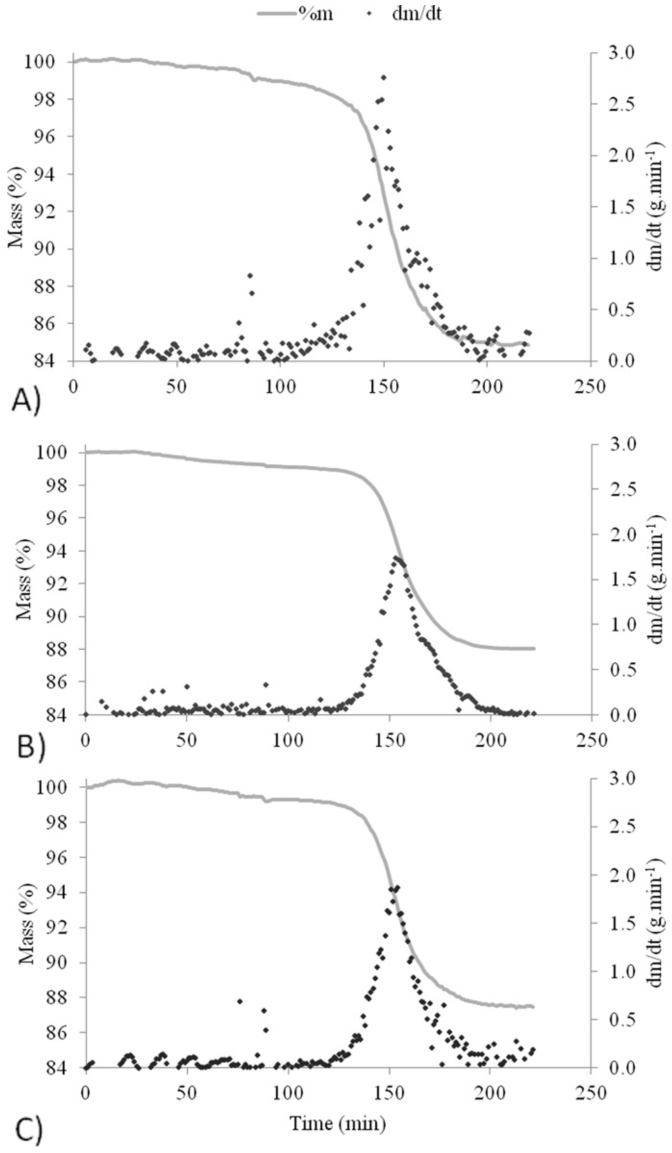

Fig. 4. Effect of *T* on mass loss and dTG for *Eucalyptus grandis* chips for each size group at 270 °C (heating rate: 4 °C min^{-1}): (A) =A; (B) =B; (C) =M.

all the more fragile and will break more easily during the mechanical shocks that typify fluidization processes. Conversely, smaller particles, <1.6 cm in our study, tend to conserve their structure without losing their resilience properties.

These results were confirmed by the Tukey test, which consists in comparing the means obtained for each study group (Fig. 5). The means before and after fluidization with the same letter did not display any significant differences at 5% probability. Only the mean of the X_3 samples displayed two distinct groups, a and b, which confirmed that the fluidization process, without prior torrefaction, indeed had an impact on the large particles by reducing their size. The results showed that fluidization had a greater impact on the non-torrefied X_1 and X_2 particles when they were isolated, i.e. they no longer belonged to a mixture as in the previous test (Fig. 5).

Table 3
Analyse of variance for the “fluidization” parameter on the particle size distribution for biomass samples from the non-torrefied group M (mixture).

Group M	R^2	*P*-value
X_0	0.480	0.513 (ns)
X_1	0.766	0.322 (ns)
X_2	0.814	0.284 (ns)
X_3	0.998	0.031 (*)

ns = not significant at 5% probability; * = significant at 5% probability.

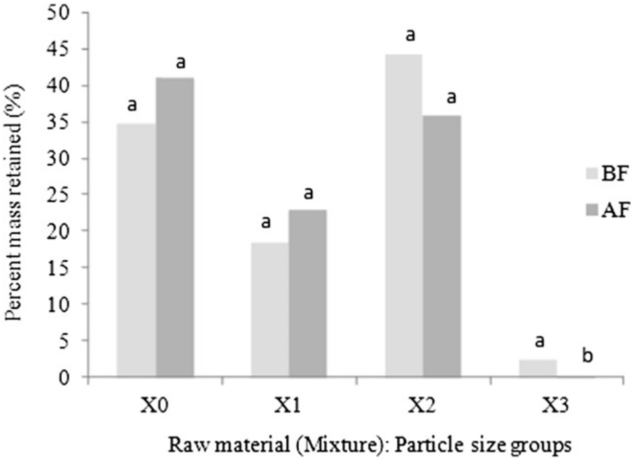

Fig. 5. Change in the particle size distribution of the non-torrefied raw material (mixture denoted M) before and after fluidization. BF = before fluidization; AF = after fluidization. For each group, the means with the same letters were not significantly different ($\alpha = 0.05$).

Tests were carried out with torrefied biomasses from groups A, B and M and the results were compared with the respective non-torrefied controls. As for calibration, the results were statistically processed. For each test, an analysis of variance and a comparison of means test were performed. Table 4 shows the results obtained for the different descriptive analysis indicators (mean, standard deviation), along with the results of the analysis of variance (R^2, *p*-value) compared to the “temperature” variable. Before torrefaction, the particle size distribution was equal to 100% of particles included between 0.28 and 0.475 cm for A and between 0.475 and 1.6 cm for B. The particle size distribution for M was as shown in Fig. 2.

Generally speaking, torrefaction had a significant impact on particle size and distribution after fluidization. A significant increase in the smallest particles was found for the three groups, and consequently a reduction in the % of the largest particles. For A and B, the reduction approached 50% in mass, with 53 and 50.1% respectively in the new redistribution. The values of the coefficient of determination, R^2, and of *p* showed that temperature was well correlated to particle size. The torrefaction temperature affected all the sizes except X_2 ($p > 0.05$) in group M, despite a 35% reduction in mass for this particle size ($0.475 < X_2 < 1.630$). The best

Table 4
Analysis of variance of the temperature (*T*) for the three response variables.

Group	Size	Control	Torrefaction/fluidization			
		Particle sizes (%)	Particle sizes (%)	Std. Dev.	R^2	*p*-values
M	X_0	34.8	51.0	10.4	0.85	0.043*
	X_1	18.6	20.5	2.2	0.84	0.043*
	X_2	44.2	28.5	8.3	0.80	0.07 (ns)
	X_3	2.4	0.1	0.1	0.94	0.006*
A	X_0	–	47.0	12.0	0.91	0.015*
	X_1	100	53.0	12.0	0.91	0.015*
B	X_0	–	28.9	10.2	0.97	0.002*
	X_1	–	21.0	2.3	0.93	0.009*
	X_2	100	50.1	10.1	0.97	0.002*

* = significant; ns = not significant at 5%.

correlations were obtained for group B ($R^2 = 0.97$), for which the new particle size distribution after fluidization was 28.9, 21 and 50.1% for X_0, X_1 and X_2 respectively. These results confirmed those obtained by Phanphanich and Sudhagar [12] who investigated the impact of torrefaction treatment on the grinding measure average particle size and size distribution. They studied torrefied pine chips and logging residues and found that smaller particle sizes are produced compared to untreated biomass. In addition, they observed that the particle distribution curve was skewed towards smaller particle sizes with increased torrefaction temperatures.

Fig. 6 shows the classification by Tukey's test of averaged results for the 4 response variables (X_0, X_1, X_2 and X_3) of group M. This comparison of means only involves particle size redistribution after fluidization before and after torrefaction. The increase in the smallest particles (X_0) was significant between the control and the biomass treated at 270 °C, with a mass increase of 35%. There was no significant difference between the biomass treated at 210 and 240 °C, or between the three treatments despite a clearly marked tendency towards an increase in small sized particles. The quantity of X_2 particles between 0.475 and 1.6 m in size decreased as the torrefaction temperature increased, though without displaying any significant difference in the means obtained. For particles of between 0.28 and 0.475 cm (X_1), the reduction was significant between the control and the treatment at 270 °C. The $X_3 > 1.6$ cm particles of group M disappeared right from 210 °C treatment. Lastly, group M, which had a particle size distribution of 40.97, 22.88, 35.86 and 0 0.28% after fluidization for X_0, X_1, X_2 and X_3 respectively, obtained a new redistribution of 63.22, 18.14 and 18.63% after torrefaction at 270 °C. Based on the data obtained with the M mixture (Fig. 6), in Fig. 7 the trends for the large particles, X_2, and for the smallest particles, X_0, were modelled using a linear equation (Table 5). This model in our temperature range (210–270 °C), or for classic torrefaction temperatures from 180 °C to 280 °C, was fairly realistic with coefficients of variation, R2, of 0.99 and 0.98 for X_2 and X_0 respectively. Several authors have tried to correlate particle size to the intensity of the heat treatment. Phanphanish [12] proposed a second order polynomial to correlate the distribution size mean particle diameter of torrefied pine chips and the torrefaction temperature (50–300 °C). This work did not investigate the fluidization of particles after torrefaction.

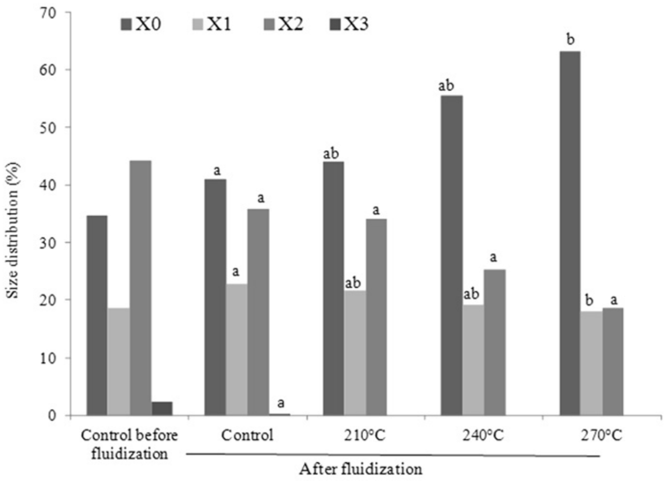

Fig. 6. Redistribution of particle sizes before and after fluidization versus temperature for the mixture group (M). The control is the untreated raw material. For each group, the means with the same letter were not significantly different at 5% ($\alpha = 0.05$).

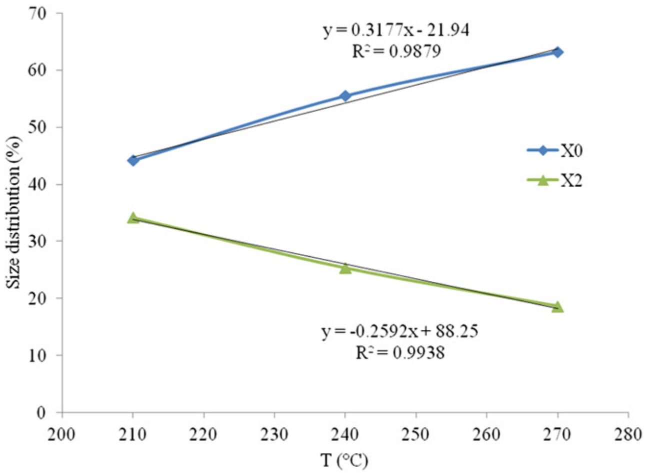

Fig. 7. Comparison of measured values and modeled values for the X_0 and X_2 particles in group M.

Fig. 8 shows the means obtained for group A after heat treatment. Before fluidization, 100% of the biomass had a particle size ranging from 0.28 to 0.475 cm (X_1). After fluidization, smaller particles were seen to appear for the non-torrefied samples with 30.8 and 69.2% for X_0 and X_1 respectively. For instance, at 270 °C, 56 and 44% were obtained for X_0 and X_1 respectively. For X_0 and X_1 the statistical study showed that the means obtained were significantly different between the fluidized control (non-torrefied sample) and the torrefied samples starting at 240 °C. At 210 °C, the differences observed no longer existed. These results confirmed the chemical modifications [22] and an alteration of the mechanical behaviour of biomass subjected to high temperatures [23,24]. Recently, Chen [25] showed that the grindability of torrefied wood could be improved in a significant way if the torrefaction temperature was as high as 250 °C and the torrefaction time longer than 1 h.

The linear models presented in Table 5 and established from the group M results were applied to group B (100% X_2 particles before torrefaction). The simulations involved X_0 and X_2 particles after torrefaction. The results are shown in Fig. 9. It can be seen that the "increase for X_0 and reduction for X_2" tendencies were similar for both the measured or calculated values. However, the measured and calculated values were significantly different, with a more pronounced difference for the high temperatures. The linear models obtained after torrefaction and fluidization of a mixture of particles (group M) were therefore not adaptable to a single group of particles such as group A or B. The initial particle size distribution of the biomass therefore had a large effect on the final distribution after torrefaction and fluidization.

The change in particle size distribution for group B (Fig. 10) displayed a similar tendency to group A. As for A, 100% of the particles had a size ranging from 0.475 to 1.6 cm. After fluidization, but without heat treatment, the new distribution revealed two new classes of smaller particles, X_0 and X_1, for which the mass proportion amounted to 15.7 and 19.5% respectively. After torrefaction, the tendency was confirmed with an increase in the smallest particles, X_0, after fluidization, whereas the X_1 particles reached a peak at

Table 5
Polynomial model for mean particle diameter of torrefied biomass, T = temperature.

Group M	Model	R^2
X_2 (0.475 cm $< X_2 <$ 1.630 cm)	$y = -0.259T + 88.25$	0.99
X_0 (<0.28 cm)	$y = 0.317T - 21.94$	0.98
Phanphanish [12]	$y = -1E^{-05}T^2 + 0.001T + 0.669$	0.99

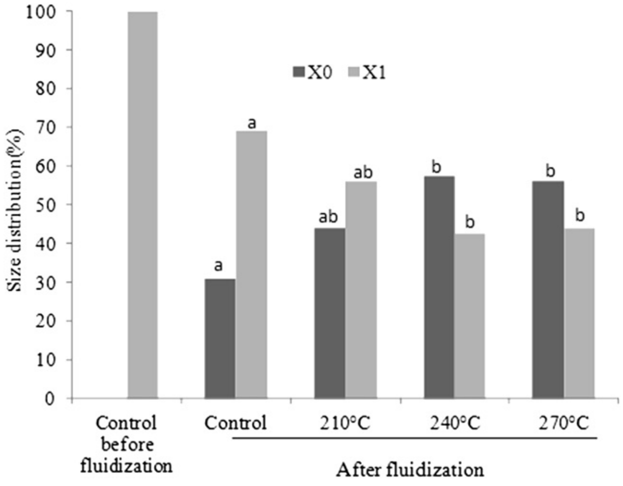

Fig. 8. Redistribution of torrefied particle sizes before and after fluidization versus temperature for group A. The control was the untreated raw material. For the two sizes X_0 and X_1, the means with the same letter were not significantly different at 5% ($\alpha = 0.05$).

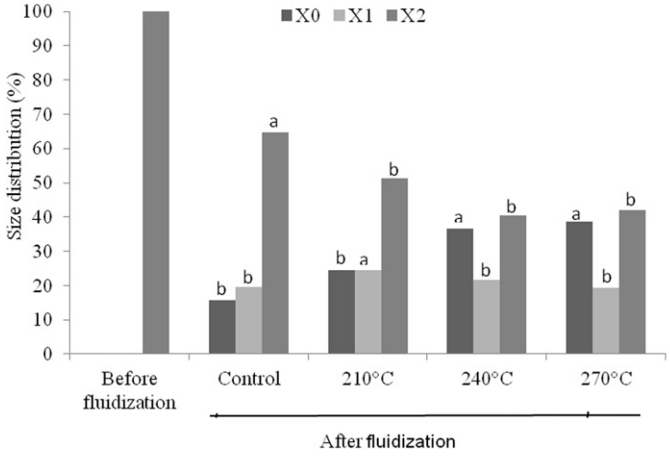

Fig. 10. Particle size redistribution before and after fluidization versus temperature for group B. The control was the untreated raw material. For the three sizes $X_{0,}$ X_1 and X_2, the means with the same letter were not significantly different at 5% ($\alpha = 0.05$).

210 °C (24.5%) then decreased beyond that temperature at 19.2% at 270 °C. It was also found that the particle size distribution was virtually the same for the treatments at 240 and 270 °C with $X_0 = 35\%$, $X_1 = 22\%$ and $X_2 = 42\%$. These results show that the resilience properties of the biomass were greatly reduced up to 240 °C, but were no longer affected even if the temperature was further increased. The relation between the process parameters and the resulting mechanical properties was examined by Poncsak [26] and he showed that mechanical strength decreases as a result of heat treatment.

The analysis of the means by the Tukey test for group B showed that there was a significant difference for the X_2 particles between the non-torrefied fluidized biomass (control) and the biomass torrefied at the three temperatures. However, no significant difference was found between the three treatments. For the smallest particles, X_0 and X_1, the results differed. For instance, for X_1, some significant differences were seen from a statistical viewpoint between the high temperatures (240 and 270 °C) and 210 °C. The same significant difference was also found between the control and 210 °C, whilst there was not difference between this control and the high temperatures. The high temperatures therefore did not lead to any change compared to the non-torrefied sample for particles between 0.28 and 0.475 cm. This can be explained by an increase in the friability of those particles during the heat treatment, leading to a displacement of the particle size distribution towards the smallest particles belonging to X_0 [27]. For the smallest particles, i.e. those under 0.28 cm, the difference was significant from 240 °C with a percentage of 37%. Below that temperature, the results did no differ from those of the control.

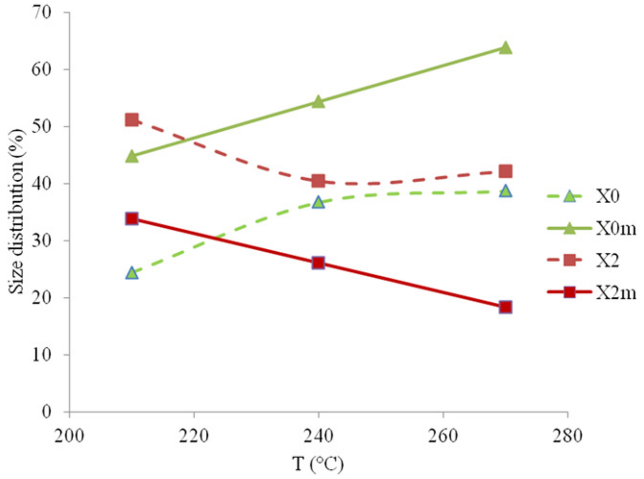

Fig. 9. Application of the group M models to group A. X_0 m and X_2 m are the values of the model.

4. Conclusion

From this work, some meaningful conclusions can be drawn:

1. Torrefaction had a significant impact on particle size and distribution after fluidization.
2. Torrefaction produced a greater mass loss for smaller particles (0.28–0.475 cm) than for particles ranging from 0.475 to 1.6 cm. Thus, the gravimetric yields were 90% and 84% for the smaller particles at 240 °C and 270 °C, respectively, and 94% and 88% for the larger ones. This may have been due to better heat and mass transfer for smaller particles.
3. For mixture M composed of all the particle sizes (cm) $X_0<0.28$, ($0.28 < X_1<0.475$, $0.475 < X_2<1.6$ and $X_3 > 1.6$, It was demonstrated that fluidization, before torrefaction, only had an impact on larger particles up to 1.6 cm. The smaller particles were not affected by their own fluidization
4. A significant increase in the smallest particles was found after torrefaction and fluidization for the three groups, and consequently a reduction in the % of the largest particles. For A (100% of X_1) and B (100% of X_2), the reduction approached 50% in mass.
5. The particle size distribution showed that torrefied biomass produces smaller particles than untreated biomass during fluidization.
6. For the mixture, particle size distribution was correlated to the intensity of the heat treatment by a single linear model in the range of 210–270 °C.

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